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Highlights

- Atmospheric/humidity cyanoacrylate fuming is superior to the vacuum process.
- Atmospheric/humidity conditions are superior for both two-step and one-step process.
- A sequence of double treatments with Lumicyano yields a higher detection rate.
- Atmospheric/humidity fuming after vacuum fuming is possible.
- Vacuum cyanoacrylate fuming may have certain operational advantages.

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A comparison between atmospheric/humidity and vacuum cyanoacrylate fuming of latent fingermarks

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Abstract

A number of pseudo-operational trials were set up to compare the atmospheric/humidity and vacuum cyanoacrylate fuming processes on plastic carrier bags. The fuming processes were compared using two-step cyanoacrylate fuming with basic yellow 40 (BY40) staining and a one-step fluorescent cyanoacrylate fuming, Lumicyano 4%. Preliminary work using planted fingermarks and split depletions were performed to identify the optimum vacuum fuming conditions. The first pseudo-operational trial compared the different fuming conditions (atmospheric/humidity vs. vacuum) for the two-step process where an additional 50% more marks were detected with the atmospheric/humidity process. None of the marks by the vacuum process could be observed visually; however, a significant number of marks were detected by fluorescence after BY40 staining. The second trial repeated the same work in trial 1 using the one-step cyanoacrylate process, Lumicyano at a concentration of 4%. Trial 2 provided comparable results to trial 1 and all the items were then re-treated with Lumicyano 4% at atmospheric/humidity conditions before dyeing with BY40 to provide the sequences of process A (Lumicyano 4% atmospheric - Lumicyano 4% atmospheric - BY40) and process B (Lumicyano 4% vacuum - Lumicyano 4% atmospheric - BY40). The number of marks (visual and fluorescent) was counted after each treatment with a substantial increase in the number of detected marks in the second and third treatments of the process. The increased detection rate after the double Lumicyano process was unexpected and may have important implications. Trial 3 was performed to investigate whether the amount of cyanoacrylate and/or fuming time had an impact on the results observed in trial 2 whereas trial 4 assessed if the double process using conventional cyanoacrylate, rather than Lumicyano 4%, provided an increased detection rate. Trials 3 and 4 confirmed that doubling the amount of Lumicyano 4% cyanoacrylate and fuming time produced a lower detection rate than the double process with Lumicyano 4%. Furthermore, the double process with conventional cyanoacrylate did not provide any benefit. Scanning electron microscopy was also performed to investigate the morphology of the cyanoacrylate polymer under different conditions.

The atmospheric/humidity process appears to be superior to the vacuum process for both the two-step and one-step cyanoacrylate fuming, although the two-step process performed better in comparison to the one-step process under vacuum conditions. Nonetheless, the use of vacuum cyanoacrylate fuming may have certain operational advantages and its use does not adversely affect subsequent cyanoacrylate fuming with atmospheric/humidity conditions.

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Keywords: Cyanoacrylate, Lumicyano, vacuum, fingermarks, enhancement, pseudo-operational trials.

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Introduction

Cyanoacrylate fuming is a routine enhancement technique for the development of latent fingermarks. When fingermark residue comes into contact with the cyanoacrylate monomer vapour, polymerisation occurs along the ridges of the fingermark to produce a white deposit [1]. Cyanoacrylate polymerisation occurs due to the reactivity of the polarised carbon to carbon double bond, which includes two electron withdrawing groups (the cyano group and the ester group). These two electron withdrawing groups make the double bond vulnerable to nucleophilic attack, therefore making the resulting anion very stable due to the negative charge being pulled across the entire molecule [2].

One mechanism for the polymerisation of cyanoacrylates suggests the formation of zwitterions with the anionic part being the active propagating species [2]. Cyanoacrylate polymerisation is base initiated and weak bases, such as water, will initiate polymer growth. The polymerisation reaction may also be accelerated by other bases such as sodium carbonate [3] and sodium hydroxide [4]. It is thought that increasing the relative humidity (RH) to 80% causes sodium chloride (NaCl) crystals in the latent fingermark to take up water. Latent residues contain other bases and some of these may also initiate polymerisation [5]. Short chains, oligomers, of cyanoacrylates may be formed due to atmospheric humidity, which could take part in further polymerisation on the fingermark [6]. Sebaceous fingermarks treated with cyanoacrylate fuming exhibit a large amount of circular polymer on the ridges as well as clumps of 'noodle-like' polymer. It is suggested that this morphology is a result of emulsion polymerisation, with fatty acids acting as emulsifiers of aqueous and oily phases. Due to the presence of the 'noodle-like' polymer in sebaceous marks, it is suggested that whatever initiates the growth of polymer in eccrine fingermarks is also present in unevenly distributed, smaller amounts in sebaceous fingermarks [6]. Lewis *et al* reported that the moisture contained within a fingermark was more important than the moisture in the air during the fuming process [7]. Eccrine fingermarks showed reduced quality of developed marks with time due to the loss of moisture from the mark. Sebaceous marks demonstrated less age dependence and it has been suggested that such marks could retain moisture in the residues over time but that the constituents of the sebaceous mark did not contribute to the polymerisation reaction [7].

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Two-step process

Following cyanoacrylate fuming, a second treatment is generally required to improve the contrast of the white cyanoacrylate polymer against the background. Currently, fluorescent dyes and powders are routinely used in these two-step cyanoacrylate processes. A methanol solution of Rhodamine 6G was proposed as a suitable fluorescent dye for cyanoacrylate polymer in the early 1980s [8,9] and is still in use in certain countries. Other countries (including the UK) consider the use of Rhodamine 6G in methanol inadvisable because of the suspected health risks posed by both dye and solvent. In 1985, the UK Home Office Centre for Applied Science and Technology (CAST, then called Police Scientific Development Branch PSDB), identified basic yellow 40 (BY40) in ethanol as a safe, effective alternative dye system to Rhodamine 6G [6]. BY40 absorbs in the violet-blue region of the light spectrum and cyanoacrylate marks treated with BY40 will emit in the green-yellow region. The use of BY40 in sequence with cyanoacrylate fuming has been shown to produce twice as many identifiable prints in comparison to cyanoacrylate treatment alone [6]. CAST trialled many other dyes, such as safranine O, ardrox and nile red, and currently recommends the use of BY40. For surfaces not compatible with ethanol or in areas with poor ventilation, a water-based formulation may be used; however, a water-based solution of basic red 14 is recommend in such instances as it produces fluorescence of higher intensity than water-based BY40 [6].

One-step process

A one-step fluorescent cyanoacrylate process combines the cyanoacrylate fuming and dyeing procedure into a single step process. This offers the possibility of saving time, space and effort as well as avoiding the use of flammable solvents. In the early 1990s, Weaver and Clary [10] reported a one-step fluorescent process using a solid cyanoacrylate polymer and 3M styryl dyes. More recently, research has investigated other one-step processes available such as Polycyano (Cyano UV, Foster and Freeman, U.K.) [11,12], fuming orange and CN yellow (Aneval, Inc., Illinois, US) [13] and Lumicyano (Crime Scene Technology, France) [14]. Most of these products require heating temperatures of $\geq 230^{o}$C with the exception of Lumicyano where a traditional hot plate temperature of 120^{o}C is required. These one-step processes appear to provide enhancement comparable to the conventional two-step process but subsequent treatment with a fluorescent dye may result in an improved detection rate as reported elsewhere [12,15,16]. The Lumicyano polymer appears to have a "slightly better developed polymeric nanofiber

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morphology in comparison with the traditional method" [17]. Furthermore, the successful tagging of cyanoacrylates with fluorescent species such as p-DMAB, p-DMAC and dansyl chloride has also been reported [17].

Atmospheric Cyanoacrylate Process

The atmospheric/humidity process involves heating the cyanoacrylate up to a temperature of 120^{o}C in a chamber at 80% RH. This results in the deposit of a white polymer along fingermark ridges where the morphology of the polymer is a long, fibrous structure which extends upwards and outwards when observed under scanning electron microscopy [6]. This 'noodle-like' polycyanoacrylate morphology allows for efficient light scattering and easier visual perception. The RH in the atmospheric process has a large influence on the development of latent fingermarks. Humidity levels that are below 75% produce underdeveloped marks and those above 80% RH tend to increase background development, therefore resulting in a reduced definition of the developed mark. The optimum RH range was reported as 85% to 90%; however, a lower value of 80% is recommended to account for the discrepancy between the fuming cabinet display and the actual relative humidity value [18]. Furthermore, it does not get too close to 100% which may result in excessive background development. Development at 60% RH yields a 'tortellini-like' polymer structure and a two-dimensional film, possibly due to the initiation by a hard anion which then leads to a very fast initiation and many active centres of polymer growth [19]. At 80% RH, the initiation of polymerisation is slower resulting in fewer active centres of polymer growth and thus leading to growth in one direction and a 'noodle-like' morphology [20]. The morphology of the cyanoacrylate at 80% RH allows for suitable visualisation due to the light scattering and because it traps fluorescent dyes molecules for successful staining and observation of fluorescence.

The atmospheric process heats up the cyanoacrylate to 120^{o}C to accelerate the fuming of marks in the cabinet; however, this may result in uneven coverage and overdevelopment where both the ridges and furrows of the latent fingermark are filled with cyanoacrylate polymer [21]. The use of high temperatures for some of the latest atmospheric one-step fluorescent cyanoacrylate processes may also result in the production of toxic hydrogen cyanide gas [22].

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Vacuum Cyanoacrylate Process

In the vacuum process, the articles to be treated are sealed in a vacuum chamber together with the cyanoacrylate. The use of the vacuum cyanoacrylate process initiated with the development of custom build chambers; however, due to high costs many other researchers utilised simpler set ups such as benchtop desiccators [23]. More recently, although not specifically designed for vacuum cyanoacrylate fuming, other low pressure chambers have been commercially developed [24,25]. Treatment pressures range from 0.1 torr to 50 Torr (1atmosphere = 760 Torr = 101325Pa = 1.013bar) [21,23,26–28] where at reduced pressure, the cyanoacrylate will vapourise at a reduced temperature and in most cases the use of assisted heating is not required, although it may be used [29]. This results in quick polymerisation due to the lack of air in the vessel, and allows the cyanoacrylate fumes to spread easily and uniformly. The negative pressure in the chamber also eliminates humidity in the tank which affects the appearance of the developed fingermarks where the polymer covers the articles with a light, even coating [23]. The morphology of the cyanoacrylate is observed as small granular beads and it does not allow suitable scattering of light; hence, it is more difficult to visualise when compared to the atmospheric/humidity process. In addition, the dye uptake may not be as effective as in the 'noodle-like' structure produced under atmospheric and 80% RH conditions. An important question to be asked here is whether the use of one-step fluorescent cyanoacrylate processes will eliminate this aspect.

The vacuum cyanoacrylate process can address a number of disadvantages from the atmospheric/humidity process. Under vacuum conditions, cyanoacrylate fuming does not cause overdevelopment but lightly covers the latent mark and generally requires fluorescent staining. Furthermore, once a vacuum is obtained, all the inner surfaces (even those not directly exposed) are treated with cyanoacrylate fumes [21]. An even coating of cyanoacrylate polymer is also observed on marks deposited on irregular or creased surfaces such as firearms and plastic bags; however, results have been found to be inconsistent and samples may have to be re-fumed [29]. Exposure to vacuum conditions may result in a significant reduction in mass (a 26% loss in mass is equivalent to around 5 weeks of ageing) and lipid composition of a latent fingermark, although this was reported for a much higher vacuum of 2×10^{-5} Torr, typically found in vacuum metal deposition systems and other vacuum-based analytical equipment [30].

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Atmospheric vs Vacuum studies

Watkin [21] performed a comparison trial of atmospheric and vacuum cyanoacrylate fuming and asked identification experts to rate the enhancement observed in a blind test. With regards to print clarity and background interference, the majority of the 54 experts participating in the study indicated the vacuum cyanoacrylate process as superior. Klasey and Barnum [31] compared the development of latent marks on firearms by vacuum and atmospheric cyanoacrylate fuming. The results demonstrated that, under vacuum conditions, the developed marks were not as 'white' which may lead to casework marks being missed. Nonetheless, the vacuum process provided superior results on blue steel surfaces suggesting it could be the method of choice for this surface. Bessman *et al* [28] performed further comparative trials and used three different types of fuming chambers - atmospheric/humidity, vacuum and a normal cabinet (one that did not use vacuum or humidity controls but just a single hot plate). The study demonstrated that both atmospheric/humidity and vacuum were superior to the normal cabinet where the atmospheric/humidity process was superior on plastic substrates and the vacuum process was superior on glass slides and Styrofoam material.

Atmospheric/humidity and vacuum cyanoacrylate processes were also studied by the UK Home Office CAST in the early 1990s using a series of pseudo-operational trials of split fingermarks on polyethylene bags [32]. Pseudo-operational trials are used to "establish whether the results obtained in laboratory trials are replicated on articles/ surfaces typical of those that may be submitted to a fingerprint laboratory, or to distinguish between closely equivalent formulations that cannot be separated in laboratory trials" [33]. For the split marks, it was reported that the intensity of observable ridge detail after dyeing was significantly lower for the vacuum process and that one-week old marks favoured atmospheric/conditions but the difference was less pronounced for one day aged marks, presumably due to higher water content after one day when compared to one week. During the pseudo-operational trial, 32 fingermarks were found with the atmospheric/humidity process compared to the 16 fingermarks with the vacuum process [32]. Another study [34] reported the opposite results where the vacuum treatment was reported as being superior with advantages such as better regularity, less coating and lower quantity of cyanoacrylate needed although the issues of low contrast are also discussed.

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Due to contradictory results concerning vacuum cyanoacrylate fuming in the literature in the early 1990s, this current work aims to carry out further comparative trials using the atmospheric/humidity and vacuum techniques with the two-step process on plastic carrier bags by means of pseudo-operational trials. The study also investigated the use of Lumicyano, a one-step cyanoacrylate process, and to date, the authors are not aware of studies reporting the use of vacuum fuming with one-step fluorescent cyanoacrylate processes. Other trials assessed the detection rate of latent fingermarks after a double process such as the sequences atmospheric-atmospheric and vacuum-atmospheric.

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Methodology

Preliminary investigation into vacuum fuming conditions

This trial used black bin liner bags (low density polyethylene) to compare the atmospheric/humidity and vacuum cyanoacrylate (CA) processes using the conventional two-step method involving the dyeing procedure with BY40. Split depletion series with up to 50 marks were set up and graded as described by CAST [33]. Other variables in the study included four ageing periods (1, 7, 14 and 28 days) and three donors (each giving a mark from the left and right hand). The atmospheric/humidity process was performed as described in the Fingermark Visualisation Manual [35] and then compared to various conditions under three different levels of vacuum (700, 50 and 5 Torr) for three different time periods (20, 40 and 60 minutes). For each depletion series, marks 1, 2, 3, 4, 5, 10, 20, 30, 40 and 50 were observed further and graded [33].

Collection of items for pseudo-operational trials

Everyday use plastic carrier bags (a mixture of HDPE, LDPE, recycled and bio), were collected at random, with no more than five bags collected from the same individual or source in an attempt to increase the variability of donors as well as the origin and type of substrates. Each trial consisted of 100 items in line with previous studies [15,16,36] and the description (e.g. colour and material type) for each item was recorded. All items were treated with the appropriate technique within three weeks of collection. The number of detected marks (visually and fluorescent) was counted at each stage of the sequence.

Trial 1

The collected plastic carrier bags were split into quarters and the opposite sides were labelled either A and B to eliminate bias as shown in figure 1. Both processes involved the conventional two-step cyanoacrylate fuming followed by BY40 staining: process A in an atmospheric/humidity chamber and process B in a vacuum chamber.

Trial 2

The collected plastic carrier bags in this trial were similarly split into quarters, with the opposite sides labelled (figure 1). Process A used Lumicyano 4% treatment in an atmospheric/humidity

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chamber followed by further Lumicyano 4% treatment in the same cabinet before staining with BY40. Process B started off with Lumicyano 4% treatment in a vacuum chamber followed by further Lumicyano 4% treatment in atmospheric/humidity chamber before staining with BY40.

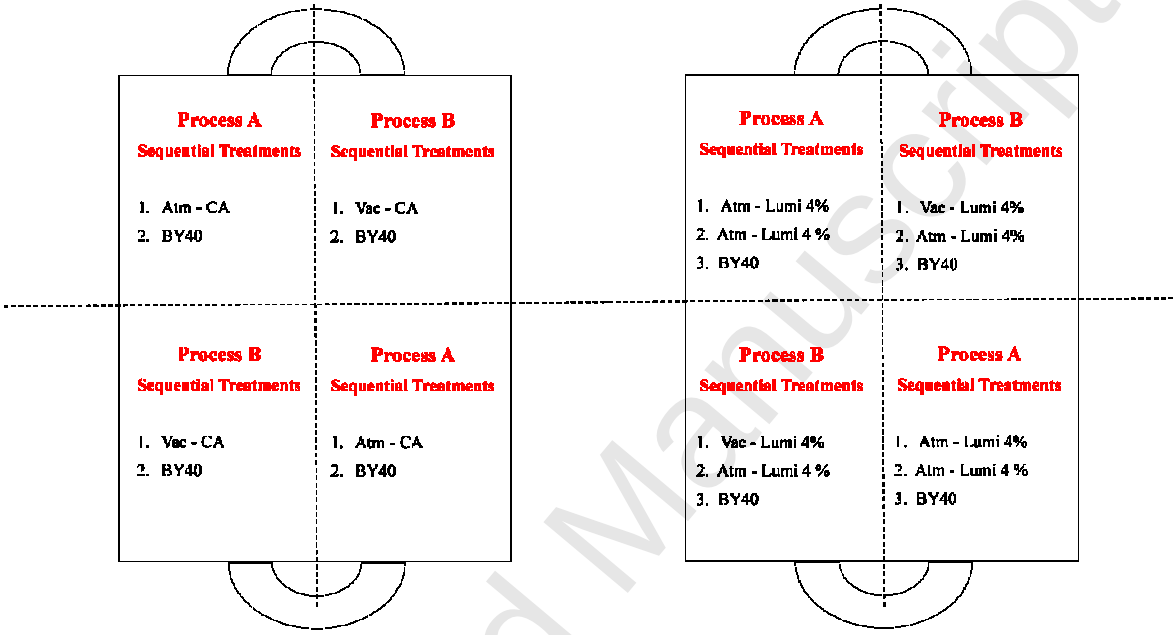

Figure 1 - Sample division for a plastic carrier bag in trials 1 (left) and 2 (right).

Trial 3

A smaller scale trial was carried out on 25 plastic carrier bags in an attempt to understand the findings from trial 2. Here, process A was repeated as in trial 2, whereas process B treated the bags at atmospheric/humidity conditions doubling the amount of Lumicyano solution/powder 4% and fuming time before finally staining with BY40 (figure 2).

Trial 4

A further smaller scale trial using cyanoacrylate (CSI Equipment Ltd.) was carried out on 25 plastic carrier bags to compare with the Lumicyano double process (atmospheric-atmospheric). Here, process A used the sequence atmospheric CA - BY40, whereas process B used the sequence atmospheric CA - atmospheric CA - BY40 (figure 2).

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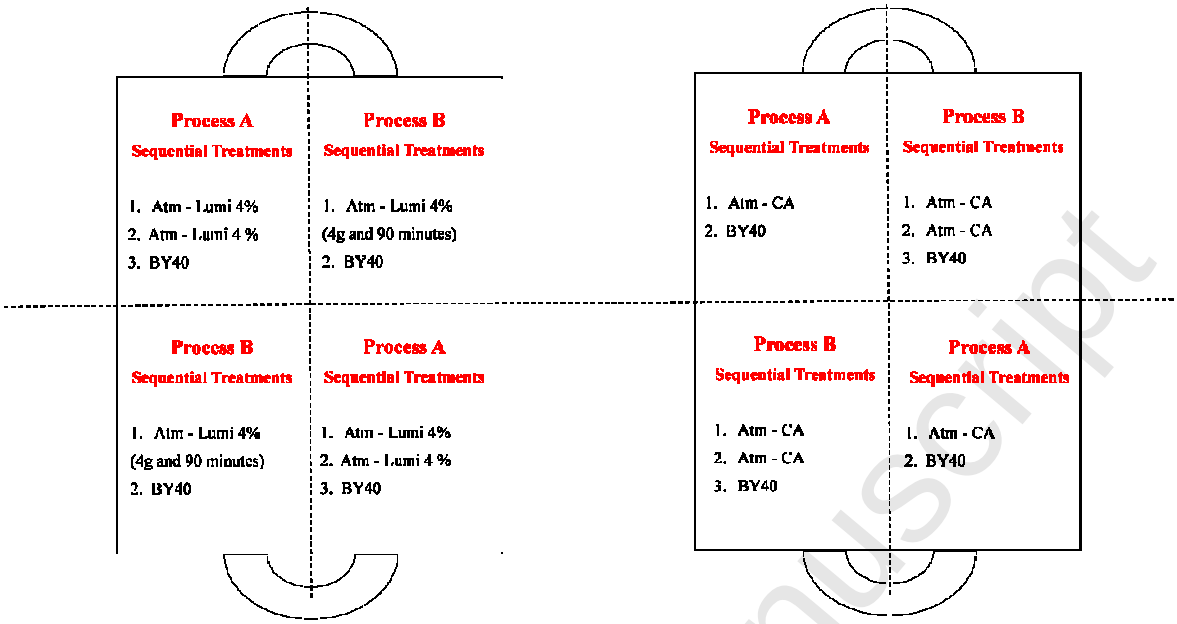

Figure 2 - Sample division for a plastic carrier bag in trials 3 (left) and 4 (right).

Atmospheric/humidity CA Chamber

An Air Science (model number CA30S) fuming chamber was employed with an approximate volume of about 450 L. The chamber is fitted with a fixed temperature hot plate (internally set to 100^{o}C) and a humidifier (set to 80%). The correct operation of the hot plate and humidifier were verified by means of a digital thermometer/thermocouple (RS 206-3738) and a humidity meter (Fluke 971).

Vacuum CA Chamber

A chamber with a volume of about 25 L and suitable of withstanding vacuum was supplied by Applied Vacuum Engineering (Bristol, UK). The air was pumped out of the chamber by means of an Edwards (England, UK) RV3 pump and the pressure measured with a BUCHI 720 pressure gauge.

Atmospheric/humidity CA [35]

2 g of cyanoacrylate (CSI equipment Ltd, UK) was placed into a new foil dish and positioned on a clean support ring on a heat source of about 100^{0}C in the fuming chamber. The relative humidity level within the chamber was set at 80% with a running time of 45 minutes. A cycle time of 45 minutes ensured that 99.99% of the cyanoacrylate had evaporated as checked by the weight difference before and after the cycle.

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Vacuum CA

0.4 g of cyanoacrylate (CSI equipment Ltd, UK) was placed into two new separate foil dishes (0.2 g x 2) and placed above the items to be fumed. The split depletion trials were carried out at three different pressures (700, 50, 5 torr) for three different time periods (20, 40, 60 minutes). The 700 torr pressure was acting as a control since it is very close to atmospheric pressure (760 Torr). The most suitable pressure and time period combination were then selected for the pseudo-operational trials.

4% Lumicyano

A 4% concentration of powder by weight of cyanoacrylate solution was prepared for both the atmospheric/humidity (0.08 g of Lumicyano Powder in 2 g Lumicyano Solution) and vacuum processes (0.008 g of Lumicyano Powder in 0.2 g Lumicyano Solution x 2) which readily dissolved to create a pink solution. The treatment procedure in both chambers was carried out as described above. After Lumicyano fuming, fluorescence was observed by exciting with a blue/green light (band pass filter 468–526 nm at 1% cut-on and cut-off points respectively) and viewed with an orange long pass 529 nm filter (1% cut-on point) followed by UV examination.

Basic Yellow 40 (BY40) [35]

After observation and photography of any marks developed by both processes in all trials, the items under examination were immersed in a BY40 solution for about a minute followed by thorough rinsing under running tap water and left to dry at room temperature before fluorescence examination. BY40 dyeing on fumed items was performed the following day after fuming. BY40 (Sirchie) dye was prepared by dissolving 2 g in 1 L of ethanol (Fisher). Fluorescence was observed by exciting with a violet-blue excitation source (band pass filter 400-469 nm at 1% cut-on and cut-off points respectively) and viewed with a yellow long pass 476 nm filter (1% cut-on point).

Photography and Fluorescence

Fluorescence examination was performed using a Mason Vactron Quaser 2000/30 and photography was carried out using a Nikon D5100 equipped with a 60 mm micro Nikon lens. UV examination was carried out using a 50 W Labino® SuperXenon Lumi Kit (peak excitation at 325 nm) and viewed with a UV face shield for UV protection.

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Evaluation of the quality of latent marks recovered in each pseudo-operational trial

Any marks developed with continuous ridge detail and an area greater than 64mm^2 were counted [33]. Each of these marks were graded 'a' for good contrast or 'b' for poor contrast and were also assessed for the presence/absence of third level detail (pore features and ridge detail). Overdeveloped marks were also noted.

Evaluation of the stability of Lumicyano fluorescence under vacuum conditions

A selection of fingermarks developed with Lumicyano under vacuum conditions was investigated further for the stability of fluorescence. Photographs of these marks were taken after 1 hour, 1 day, 2 days and 7 days after development. Half of each sample was stored in a sealed Kraft envelope at room temperature in a cool, dry and dark cupboard and the other half left on an open bench for the same period of time. The representative samples were then re-fumed with Lumicyano under vacuum conditions followed by subsequent BY40 dyeing.

SEM analysis of developed marks

Fingermarks developed on a variety of substrates, including low and high density polyethylene, metallised plastic films and other packaging materials, were analysed using secondary electron imaging scanning electron microscopy (SEI SEM). 29 representative samples were collected from fingermarks (not planted) successfully developed with each chosen fuming method for evaluation and comparative purposes, as outlined in Table 1 below.

Table 1 – SEM analysis: development conditions and number of samples.

Development conditions	**Samples analysed**
(1) Cyanoacrylate BY40 atmospheric (Trial 1 – Process A)	5
(2) Lumicyano 4% atmospheric (Trial 2 – Process A)	6
(3) Cyanoacrylate BY40 vacuum (Trial 1 – Process B)	6
(4) Lumicyano 4% vacuum (Trial 2 – Process B)	6
(5) Lumicyano 4% atmospheric – Lumicyano 4% atmospheric - BY40 (Trial 2 – Process A)	3
(6) Lumicyano 4% vacuum – Lumicyano 4% atmospheric - BY40 (Trial 2 – Process B)	3

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Only well-developed marks showing over $^{2}/_{3}$ of ridge detail were selected for analysis. Samples were mounted on aluminium stubs using a carbon-loaded pressure sensitive adhesive. They were subsequently coated with a ~1 nm thick layer of uniformly deposited platinum to increase surface conductivity and prevent electron charge build-up during analysis. Imaging was carried out in a Zeiss Supra 35VP field emission scanning electron microscope (FEG-SEM). This was operated in high vacuum at a low voltage of 3 kV and a working distance of 5 mm for effective visualisation of the polymerised developer, precluding beam damage to any surface features.

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Results and Discussion

Preliminary work into vacuum conditions from deposited marks

The data from the split depletion trials indicated that vacuum cyanoacrylate fuming may be a viable enhancement technique even after the use of a stain despite previous reports stating that is less effective [32]. BY40 staining of marks developed under vacuum conditions was sometimes successful and on other occasions it was not. Figure 3 compares the enhancement obtained by atmospheric/humidity and vacuum fuming followed by BY40 staining on marks 9 and 10 in a depletion series as observed with a violet-blue light and yellow filter. Some of the marks developed under atmospheric/humidity conditions could be observed under white light; however, none of those developed under vacuum conditions could be observed until staining with BY40 and fluorescence examination. On numerous occasions, it was possible to develop up to mark number 50 in the depletion series under atmospheric/humidity conditions, although this was dependant on the donor and ageing period. The sensitivity was less under vacuum conditions, more so with the increase of the ageing period in line with previous research [32]. Increased background staining under vacuum conditions was observed and could be a result of the fact that under such conditions the cyanoacrylate polymer is delivered uniformly across the substrate. This does not hinder the observation of third level detail of the developed mark.

Treatment pressures reported in the literature for vacuum cyanoacrylate fuming can range from 0.1 torr to 50 Torr [21,23,26–28] and expensive gauges may be required to accurately measure pressure below 1 Torr. The results from the split depletion trials indicated that the optimum conditions for vacuum fuming are 5 Torr for a period of 40 minutes. The fuming time of 20 minutes was insufficient for effective development and although 60 minutes fuming time did not appear to damage the latent mark, an extended exposure did not improve on the development seen with shorter fuming times of 40 minutes. As expected, the residual cyanoacrylate in the foil dish at 700 Torr was higher than at 5 Torr where the average percentage of CA used after 40 minutes fuming at 700 Torr and 5 Torr was 11% and 97% respectively.

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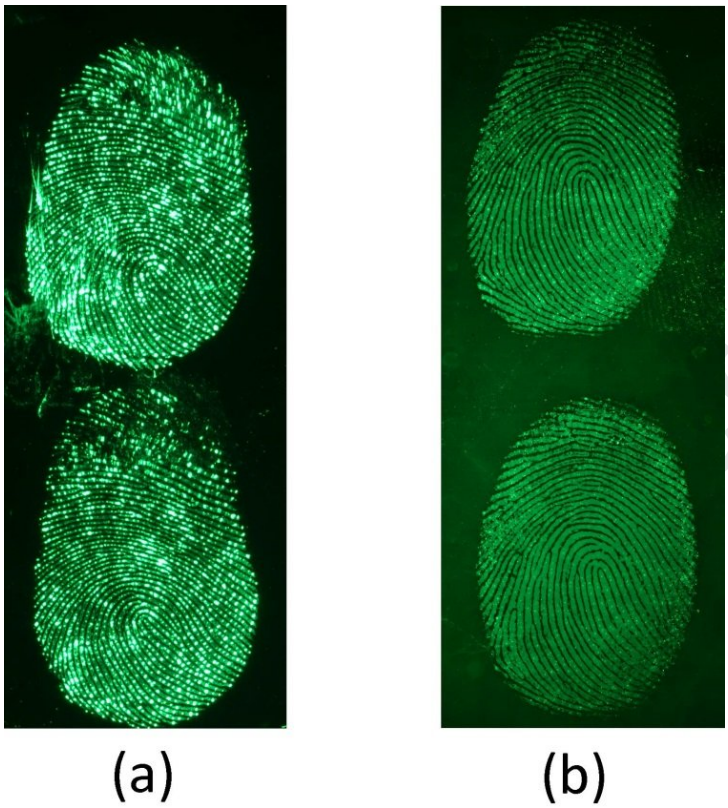

Figure 3 – Marks 9 and 10 in the depletion series after 1 day ageing developed by cyanoacrylate fuming followed by BY40 staining and viewed with a violet-blue light (yellow filter): (a) atmospheric/ humidity process; (b) vacuum process.

Figure 4 shows a split depletion series for donor 1 using the atmospheric/humidity and vacuum fuming techniques followed by BY40 staining. For atmospheric/humidity conditions, ridge detail was occasionally visible up to the 50th depletion whereas vacuum conditions only provided visible ridge detail up to the 5th mark under white light. This preliminary trial indicated that atmospheric/humidity conditions for the two-step cyanoacrylate process is superior to vacuum fuming in terms of the quality of the marks as well as the sensitivity of the technique down the depletion series and with the increase of the ageing period. This trial demonstrated that (1) the preferential conditions for vacuum fuming in the trial were 5 Torr for 40 minutes and (2) there were double the number of marks graded 3 or 4 by the atmospheric/humidity process compared to those developed by the vacuum process. Nonetheless, it was believed that pseudo-operational trials on plastic carrier bags using the two-step process and the one-step process of Lumicyano 4% may provide further insight into the vacuum process.

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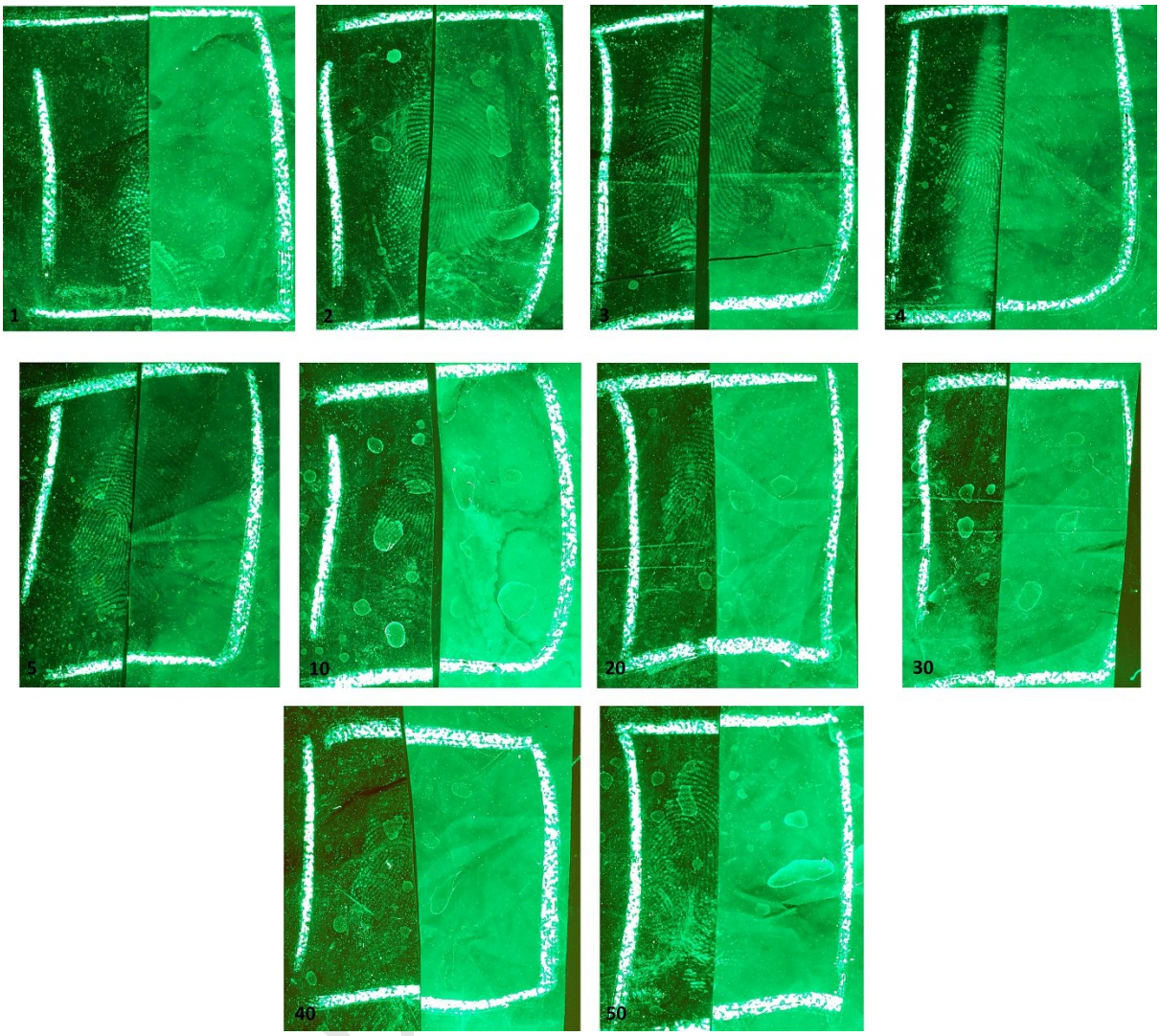

Figure 4 – Development of latent fingermarks after 7 days ageing on a black bin bag with cyanoacrylate followed by BY40 staining and observed under violet-blue light (yellow filter) down the depletion series (1,2,3,4,5,10,20,30,40,50) for donor 1 under atmospheric/humidity conditions (left) and vacuum conditions (right).

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Trial 1 (Atmospheric/humidity vs vacuum CA/BY40)

Figure 5 summarises the number of latent marks detected in trial 1 by each enhancement process as observed visually under white light and under fluorescent lighting (violet-blue light and yellow filter). Although many marks could be observed under white light when enhanced with the atmospheric/humidity process, it was easier to observe the developed marks with fluorescence. About 25% of the marks developed under atmospheric/humidity conditions were only detected with the aid of fluorescence. There was a 50% increase to the number of marks developed under atmospheric/humidity conditions when compared to vacuum conditions after treatment with BY40. As expected, no marks were observed visually when developed under vacuum conditions, owing to the cyanoacrylate deposition morphology observed under such conditions not allowing for suitable scattering of light (see SEM analysis below). Nonetheless, after treatment with BY40 and fluorescent lighting, 250 marks were observed.

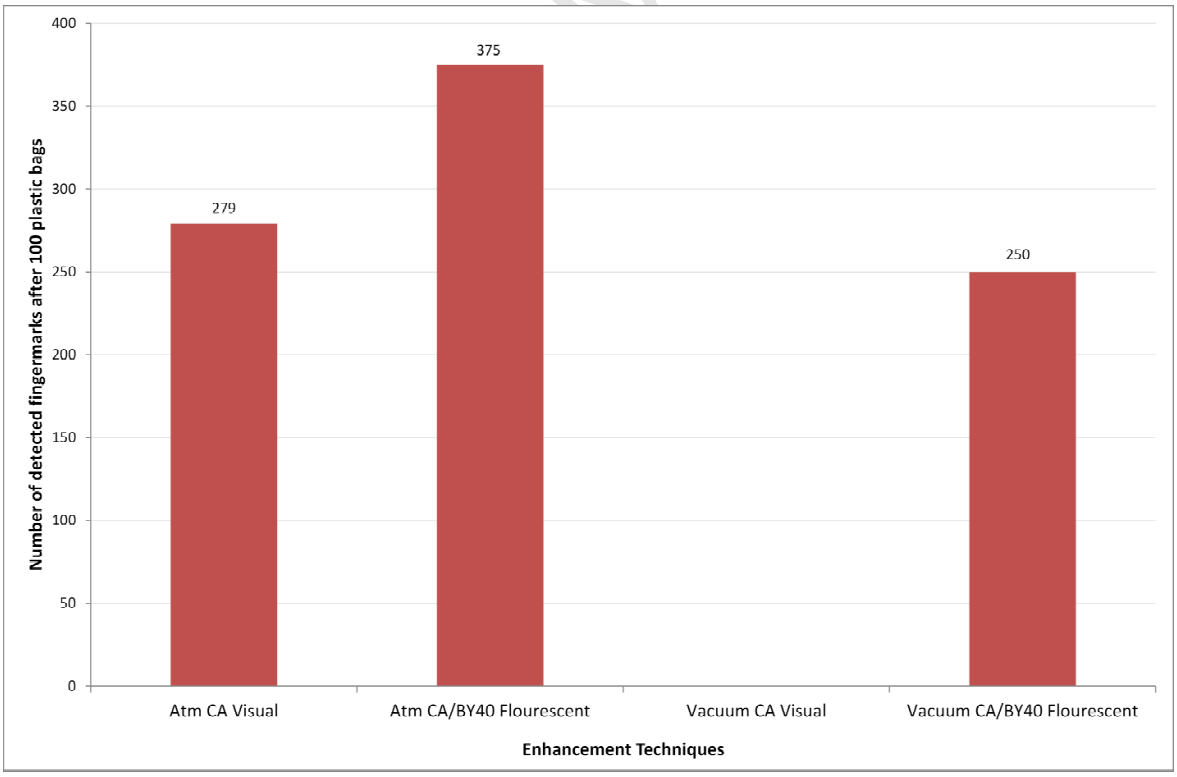

Figure 5 - Number of detected latent fingermarks for each enhancement process in trial 1.

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Trial 2

Figure 6 represents the number of marks detected by the individual treatments in process A (Lumicyano 4% atmospheric - Lumicyano 4% atmospheric - BY40) in trial 2. The use of a blue-green light (orange filter) provided better contrast and visualisation than UV light for the observation of Lumicyano 4% fluorescence. The additional number of marks by the second treatment in process A (Lumicyano 4% atmospheric) was unexpected; however, the increased detection by the third treatment (BY40) was expected as observed in other trials [15,16]. The increased detection rate by the second treatment did not result in over-fumed marks and may be explained by the fact that the cyanoacrylate polymer in Lumicyano grows in the z-direction above the ridges, rather than in the x-y plane [12, 37]. After the first treatment in process A, some marks may have been weakly developed that are not readily observed by visual and/or fluorescence examination. The second treatment resulted in further Lumicyano cyanoacrylate deposition on these weak marks enabling them to be detected in the second examination. The use of subsequent BY40 staining resulted in a further increase in the number of marks. Marks developed by the first treatment in process A were not affected by the subsequent second and third treatment (figure 7). Comparing the first treatment in process A (Lumicyano 4% atmospheric/humidity conditions) in trial 2 maps out very similarly to process A in trial 1 (cyanoacrylate/BY40 atmospheric/humidity conditions).

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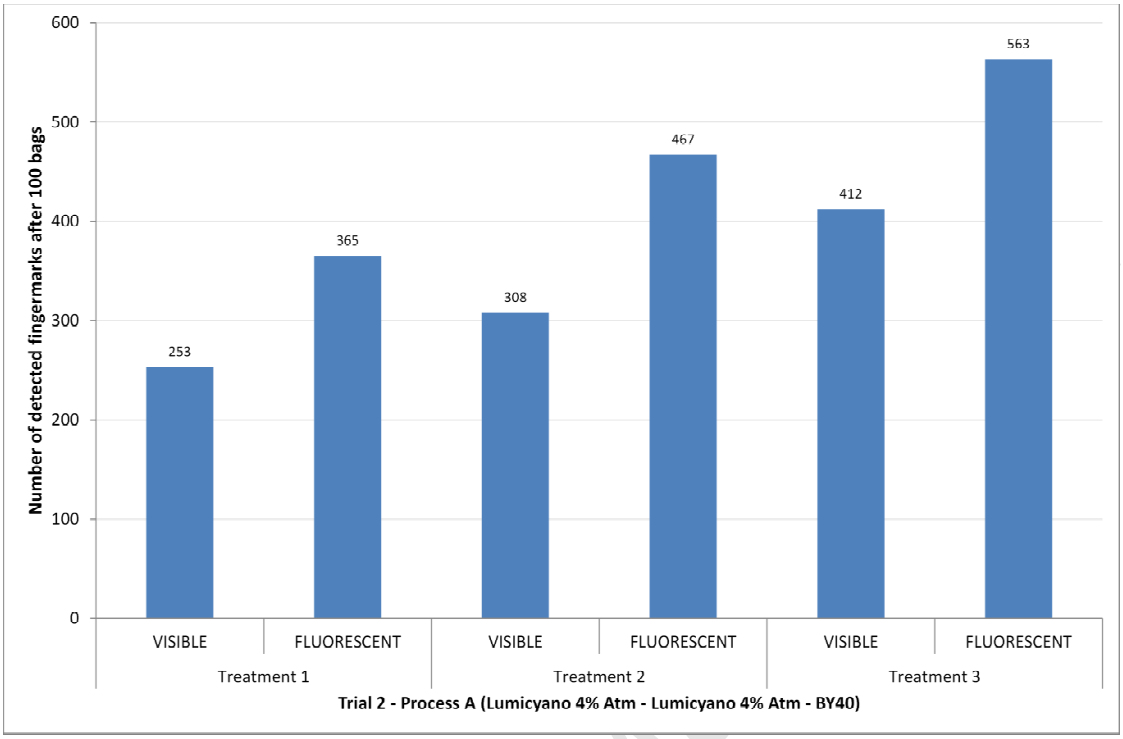
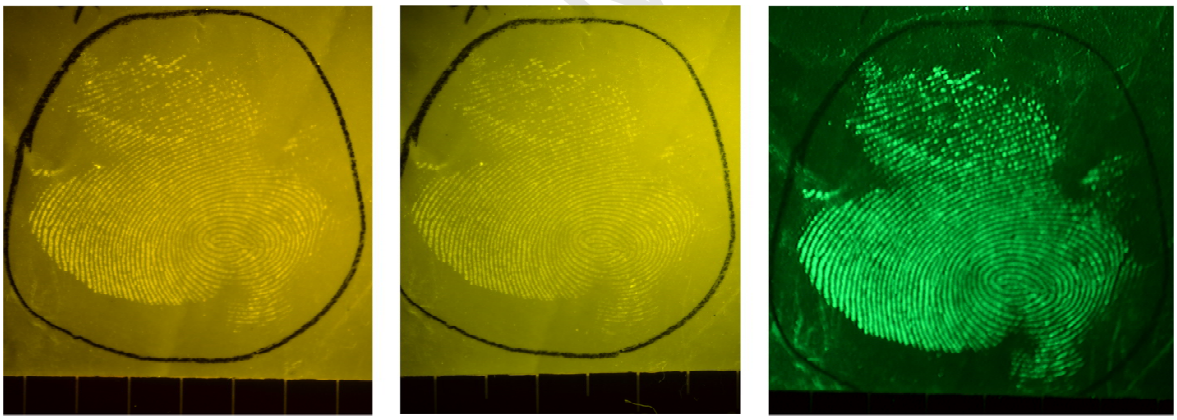


Figure 6 - Number of detected latent fingermarks for Process A in trial 2.



(a) (b) (c)

Figure 7 – Latent mark enhanced by different treatments in process A of trial 2: (a) Lumicyano 4% atmospheric/humidity observed under blue-green light (orange filter); (b) further treatment with Lumicyano 4% atmospheric/humidity observed under blue-green light (orange filter); (c) subsequent BY40 staining observed under violet-blue light (yellow filter).

Figure 8 summarises the number of marks observed by each treatment in process B (Lumicyano 4% vacuum - Lumicyano 4% atmospheric - BY40) of trial 2. The end result in process B of trial 2 (522 marks) was similar to the end result of process A (563 marks); however, the first treatment (Lumicyano 4% vacuum) of process B only provided a small number of marks. The

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second treatment (Lumicyano 4% atmospheric) in process B resulted in a substantial increase from 81 detected marks to 382 (372%). This may be explained as for process A where the Lumicyano cyanoacrylate polymer grows in the z-direction. Furthermore, the morphology of the cyanoacrylate polymer changes from small granular beads (vacuum) to a 'noodle-like' structure (atmospheric/humidity) which then responds very well to light scattering and uptake of the BY40 dye (see SEM analysis below). Although only a small number of marks were detected by the vacuum process, it did not affect the development of new marks with subsequent atmospheric/humidity conditions. Furthermore, marks developed by the first treatment in process B under vacuum conditions were not affected by the subsequent second and third treatments (figure 9). Both figures 7 and 9 show different hues for images a) and b) although the same lighting and filtration has been used. This may be explained by the fact that additional Lumicyano product is deposited in the second treatment which results in a change of hue. Furthermore, for figure 9, the morphology of the cyanoacrylate changes from beads (vacuum conditions) to a noodle-like structure (atmospheric/humidity conditions) which affects the scattering of light during visualisation and photography.

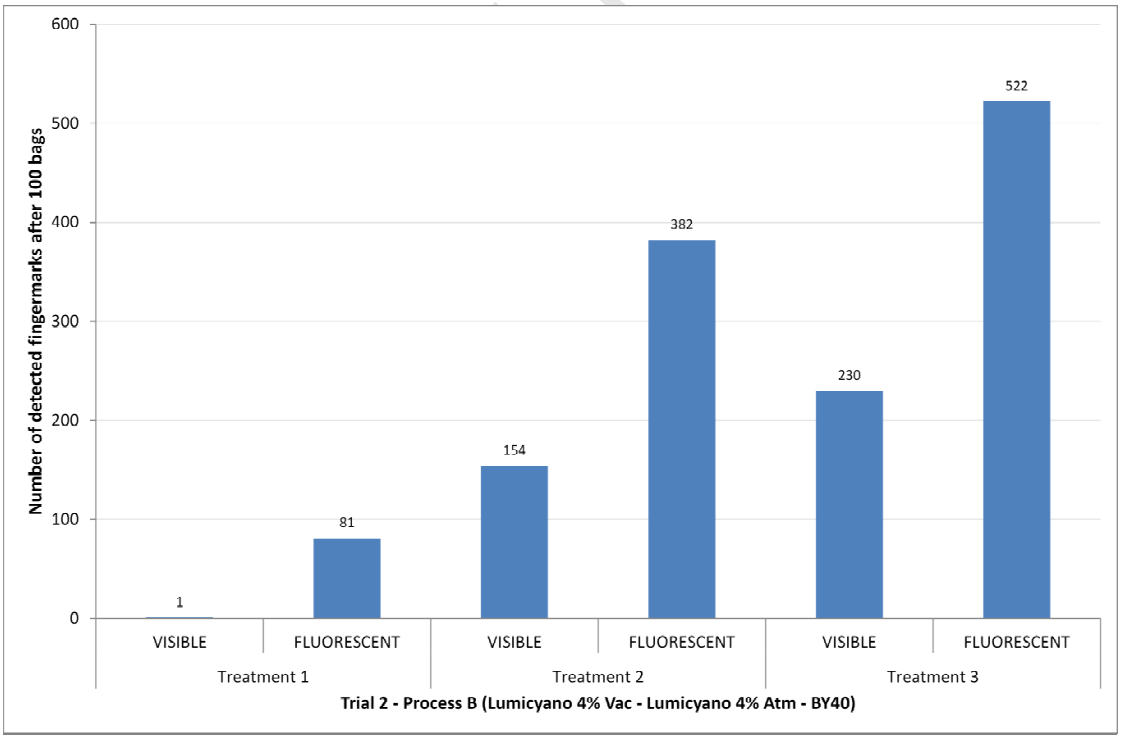

Figure 8 - Number of detected latent fingermarks for Process B in trial 2.

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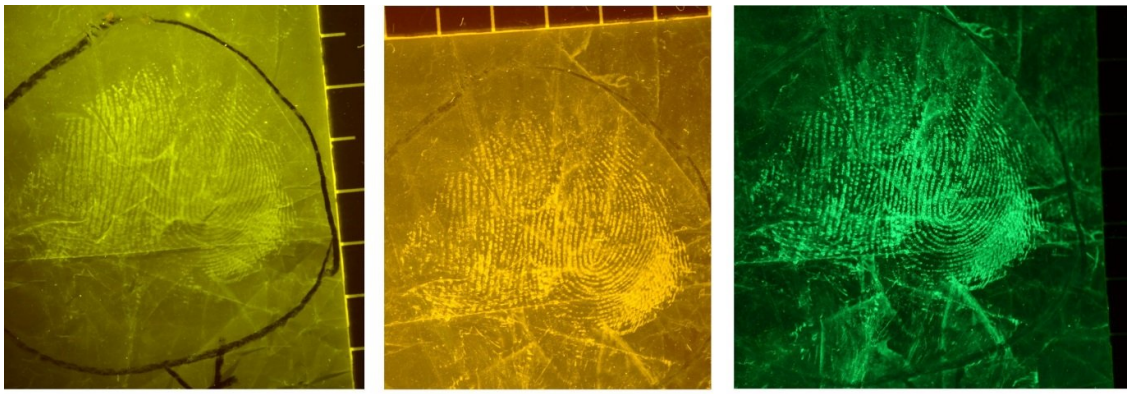

Figure 9 – Latent mark enhanced by different treatments in process B of trial 2: (a) Lumicyano 4% vacuum observed under blue-green light (orange filter); (b) further treatment with Lumicyano 4% atmospheric/humidity observed under blue-green light (orange filter); (c) subsequent BY40 staining observed under violet-blue light (yellow filter).

Other work in this study (not in this pseudo-operational trial) investigated the use of BY40 directly after Lumicyano treatment under vacuum conditions and the results varied in terms of whether the treatment with BY40 was detrimental or not. Figure 10 shows an example of where the effect observed was detrimental to the original mark. This is contrary to what was observed for marks enhanced with standard cyanoacrylate under vacuum conditions and subsequent BY40 staining (figure 3). On other occasions, the use of BY40 did not affect the previous Lumicyano 4% enhancement obtained under vacuum conditions.

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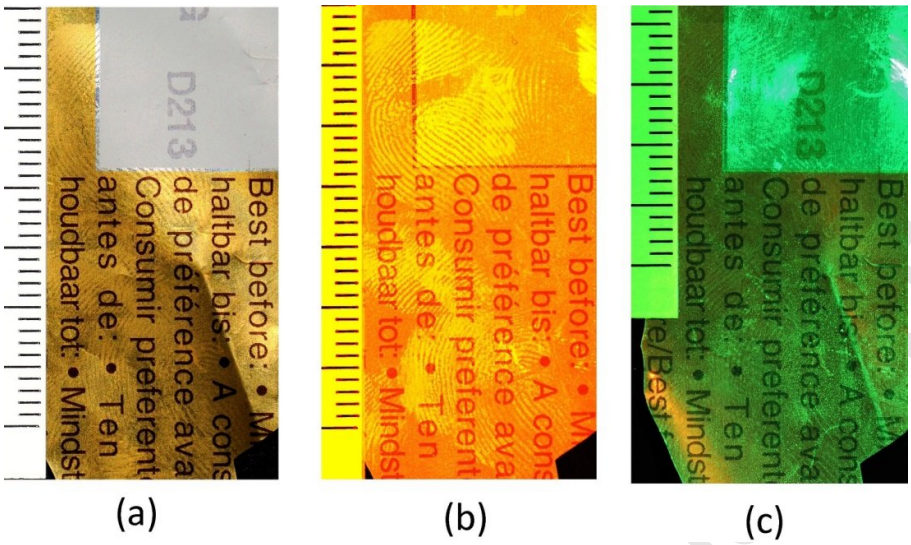

Figure 10 – Latent mark enhanced on a chocolate wrapper with Lumicyano 4% under vacuum conditions: (a) observed under white light; (b) observed under blue-green light (orange filter); (c) subsequent BY40 staining observed under violet-blue light (yellow filter).

The use of the vacuum process may have certain operational advantages in cases where the evidence is not directly exposed to the cyanoacrylate fumes. Preliminary work has demonstrated that under vacuum conditions, marks can still be developed on plastic bags/items sealed in another plastic bag (figure 11), and on CDs/DVDs stacked on top of each other. An operational example may include drugs packaging where the action of unwrapping one layer may damage fingermarks on further layers below.

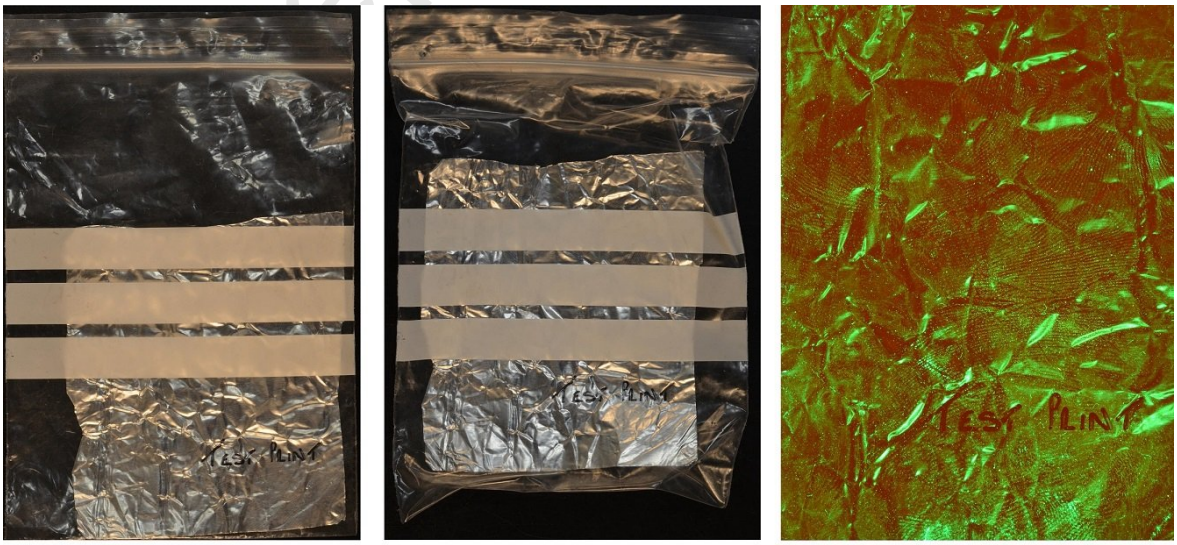

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Figure 11 – Enhanced ridge detail on foil wrapping sealed in a plastic bag under vacuum conditions: (a) under white light prior to the vacuum process; (b) under white light after the vacuum process; (c) after the vacuum process and BY40 staining observed under violet-blue light (yellow filter).

Trial 3

This trial was carried out to gauge whether the amount of glue and fuming time played a role in the number of detected marks after the second treatment in process A of trial 2 (Lumicyano 4% atmospheric - Lumicyano 4% atmospheric – BY40). This trial was executed on a smaller sample set of 25 plastic carrier bags in an attempt to understand why so many more marks were being detected after the second treatment in process A of trial 2. Figures 12 and 13 summarise the number of detected marks by each treatment of process A and B respectively in trial 3. Process A provided close to 50% additional marks when compared to process B, suggesting that the amount of glue and fuming time are not a factor for the high number of marks observed in the sequence Lumicyano 4% atmospheric - Lumicyano 4% atmospheric in trial 2. The break in the two fuming cycles appears to be an important factor and can be explained by the polymer growth of Lumicyano cyanoacrylate in the z-direction as well as the fact that Lumicyano can target cyanoacrylate deposits for the polymer growth; hence, the marks remaining undetected from the first treatment may be acting as an activation point for the polymer growth in the second treatment. Process B did not demonstrate any signs of over-fuming even though the amount of glue and fuming time were doubled.

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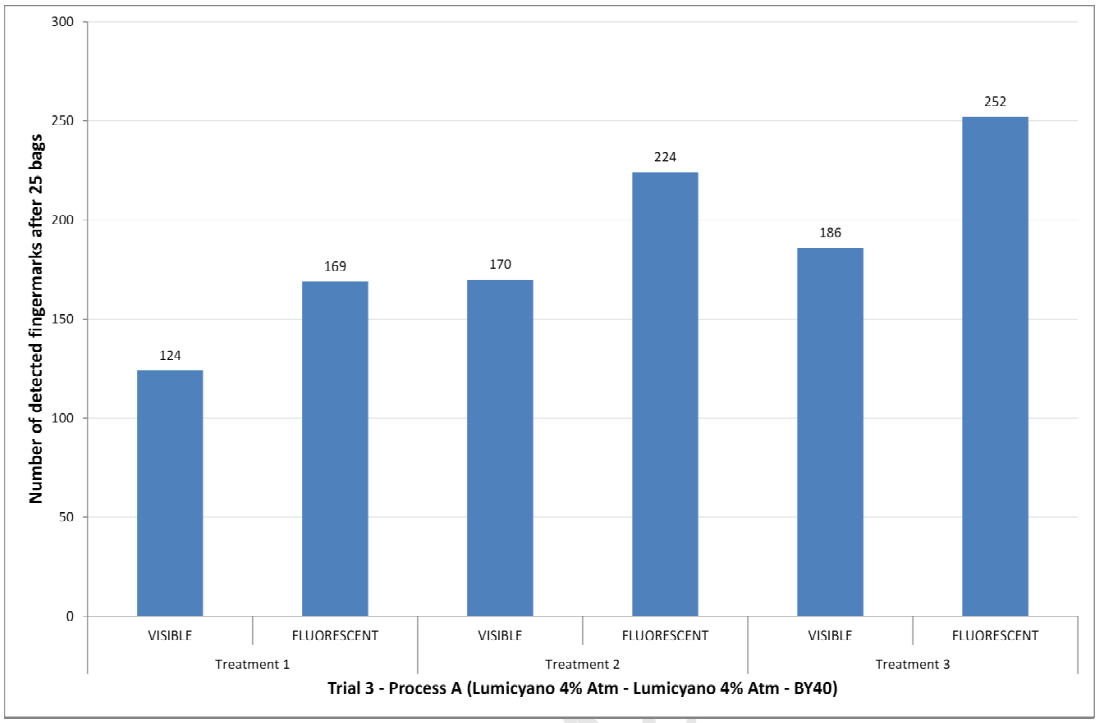

Figure 12 - Number of detected latent fingermarks for Process A in trial 3.

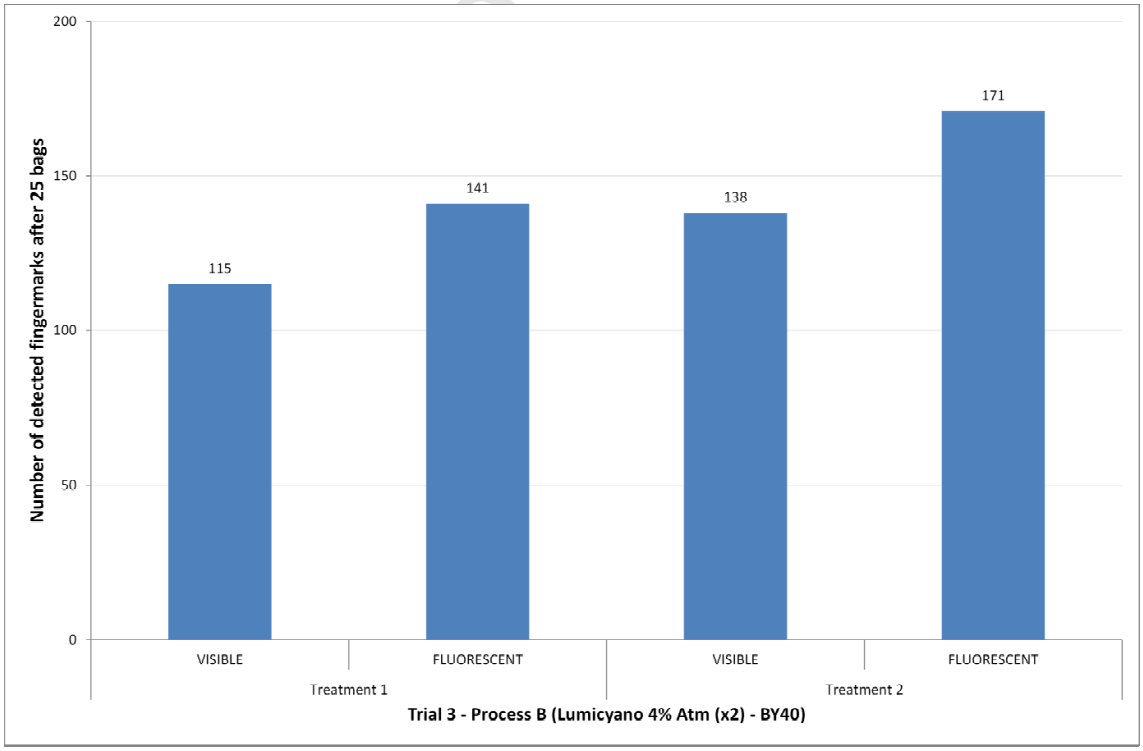

Figure 13 - Number of detected latent fingermarks for Process B in trial 3.

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Trial 4

This trial was performed to understand if the growth of the Lumicyano polymer in the z-direction is observed in the conventional cyanoacrylate used for two-step processes. The number of marks from both process A and B was very close with no apparent gain from the double fuming treatment (table 2). Although an additional 4 latent marks were detected with the double fuming treatment (Process B), this is insignificant when compared to the Lumicyano double treatment. Nonetheless, marks detected from the first treatment were not over-fumed when subjected to the second fuming treatment.

Table 2 – Cumulative number of latent marks detected by each process in trial 4.

Process A	No. of marks	Process B	No. of marks
CA-Atm Visible	60	CA-Atm Visible	67
BY40 Fluorescence	74	CA-Atm Visible	71
		BY40 Fluorescence	84

Evaluation of the stability of Lumicyano fluorescence under vacuum conditions

A selection of fingermarks developed with Lumicyano under vacuum conditions was investigated further to assess fluorescence decay over time and storage conditions. The manufacturer's guidelines state that examination and photography should take place as soon as possible. Previous studies had demonstrated that, under atmospheric/humidity conditions, the Lumicyano fluorescence had decayed completely after 1 week at a concentration of 1% [15] but lasted for up to 4 weeks at a concentration of 4% [16]. In this study, Lumicyano fluorescence at a concentration of 4% under vacuum conditions had significantly decayed after 1 day and was completely decayed after 7 days, irrespective of whether the mark was stored under daylight or dark conditions (figure 14c). The faster decay of Lumicyano 4% under vacuum conditions, as compared to under atmospheric/humidity conditions, may be due to the difference in cyanoacrylate morphology resulting in poor uptake of the Lumicyano dye. It was possible to restore or strengthen the fluorescence by re-fuming with Lumicyano 4% under vacuum conditions (figure 14e); however, it was not always as bright as the original 1 hour samples (figure 14b). Further treatment with BY40 (figure 14f) resulted in the significant deterioration of ridge detail, although this was not always the case. Manipulation with computer software of the acquired images is likely to enhance the fluorescence in figure 14 further. None of the images presented in this study have been enhanced with computer software to improve fluorescence.

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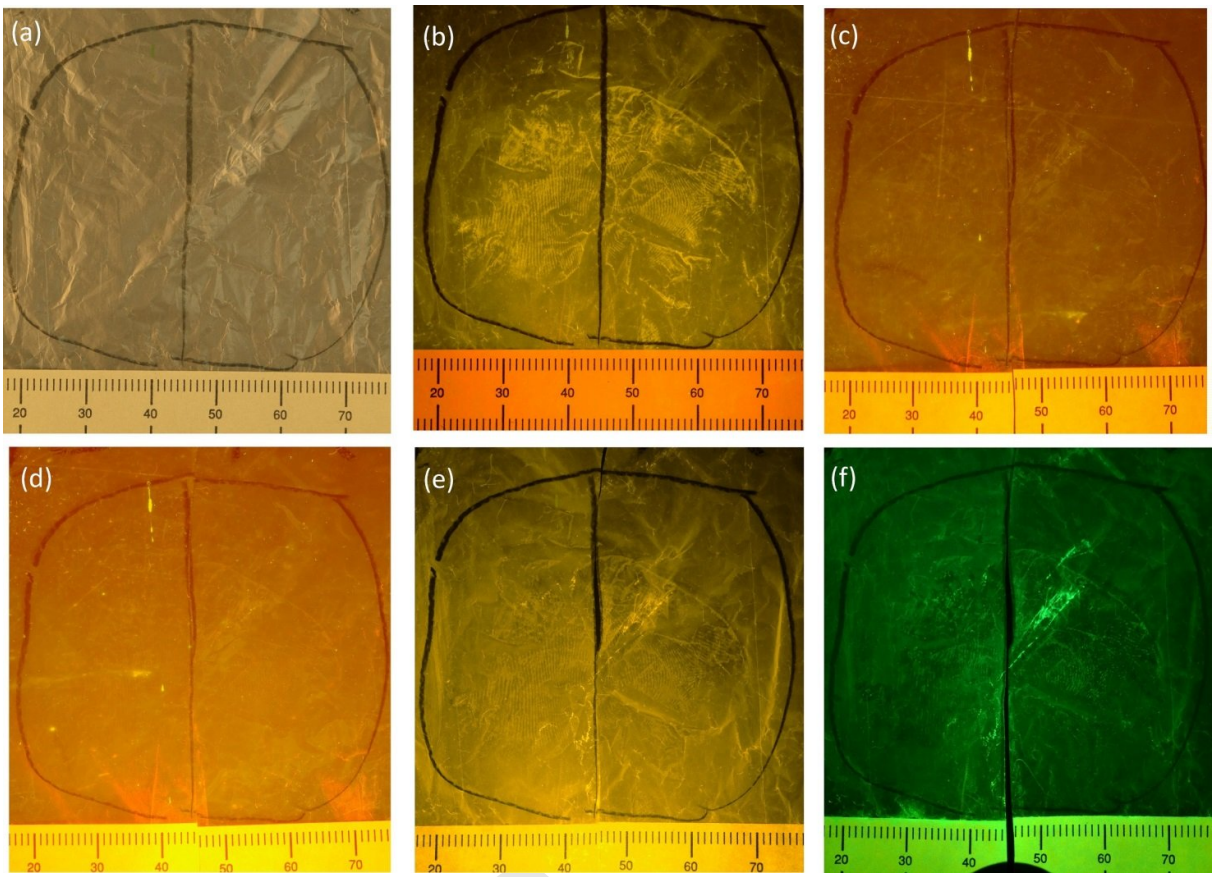

Figure 14 – A fingermark on a plastic carrier bag treated with Lumicyano 4% under vacuum conditions [left part stored in the dark, right part stored on open bench] under (a) white light; (b) blue/green (BG) light (orange filter) within an hour of fuming; (c) BG light (orange filter) after 1 day (d) BG light (orange filer) after 1 week followed by (e) re-fuming with Lumicyano 4% under vacuum conditions after 1 week [BG light (orange filter)] and (f) sequential BY40 treatment of (e) [violet/blue light (yellow filter)]

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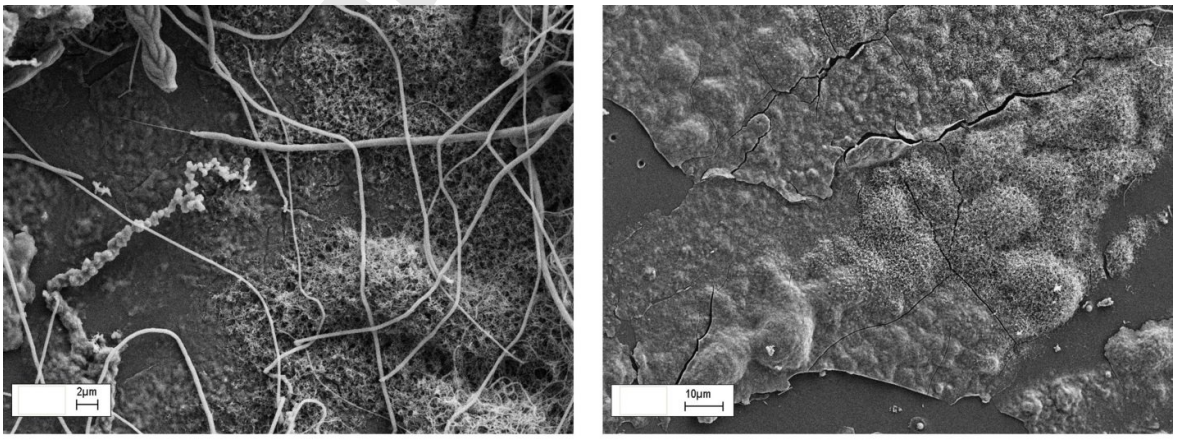
SEM analysis

(1) CA/BY40, atmospheric (Trial 1 Process A)

Polymerisation of cyanoacrylate generally manifested either as a series of 'noodle-like' fibrous structures which extended upwards and outwards or as granule-like formations. There appeared to be a tendency for fewer noodles to develop on oilier surfaces; such as the inner surfaces of crisp packets. These substrates showed a polymer build-up into a more granular-like morphology, exhibiting a series of peaks and troughs over the ridges with spheres that fused in part into a more two-dimensional film (Figure 15). Some fibrous 'noodles' that varied in density and abundance were also present but, generally, constituted less than ~5% of the developed fingerprints. Examination of samples under higher magnifications of ×20,000 – 50,000 however revealed the presence of much smaller, short and dense microfibril structures, which suggested a varied polymerisation process over the oily surface. Polymerisation of the cyanoacrylate on 'cleaner' surfaces, such as the polyethylene (PE) plastic bags, resulted in large accretions of haphazard arrays of islands of long 'noodle-like' fibrous structures that covered ~10% of the developed print (Figures 16a, 17a). The remaining area on these surfaces, as with the oily prints, polymerised into a more granular two-dimensional structure. Spherical accretions in these samples were dense, but remained separate/partially-fused with particle-size varying between ~0.5-1.5 μm in size (Figure16b).



Figures 15 - SEM images from two different fingermarks on the same crisp packet after enhancement with CA/BY40 at atmospheric conditions: (a) A range of large noodles and smaller fibrils and globules (×7K); (b) the more uniform, granular, polymer film, with short microfibrils also apparent on part of the polymerised surface (×2.5K).

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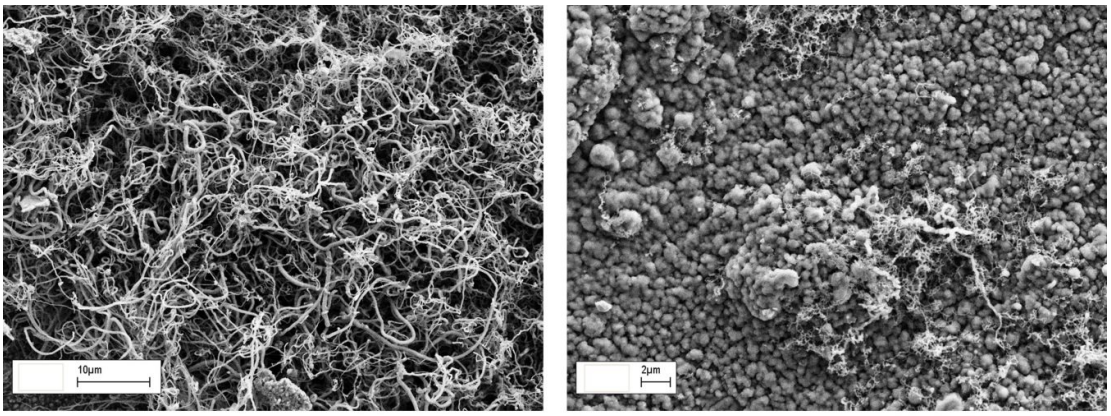

Figures 16 - SEM images from different areas of the same fingermark on a black bin bag after enhancement with CA/BY40 at atmospheric conditions: (a) An example of a well-defined mass of large noodles (×4K); (b) the largely separate spherical polymer structure on the surface, with a scattered array of small fibrils (×10K).

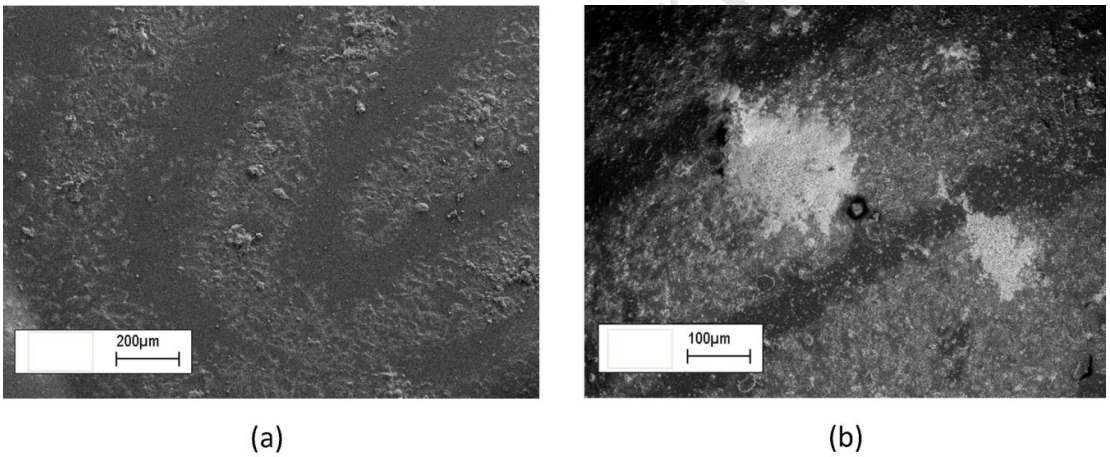

Figure 17 - Noodle accretions on the developed fingerprint after enhancement with CA/BY40 at atmospheric conditions on: (a) an interior of a metallised plastic film (crisp packet ×150); (b) a PE surface (×250).

(2) Lumicyano 4%, atmospheric (Trial 2 Process A 1st treatment)

SEM images of fingermarks developed with Lumicyano under atmospheric pressure exhibited a less-obvious division between 'clean' and 'oilier' prints. Closer examination of lipid-rich substrates (crisp packets) showed that fibrils on these surfaces appeared finer and denser, and in certain samples also exhibited nodular growths, with spheres of condensed polymer of ~3 µm diameter attaching to the fibrils (Figure 18b). In most samples the polymer condensed into long, 'noodle-like' fibrous formations, which were similar in structure to those observed with cyanoacrylate-BY40 (atmospheric). These, however, covered the entire ridge detail; therefore strongly suggesting that Lumicyano preferentially condenses as long growths of noodles on fingermarks when fumed under atmospheric conditions (Figure 18a).

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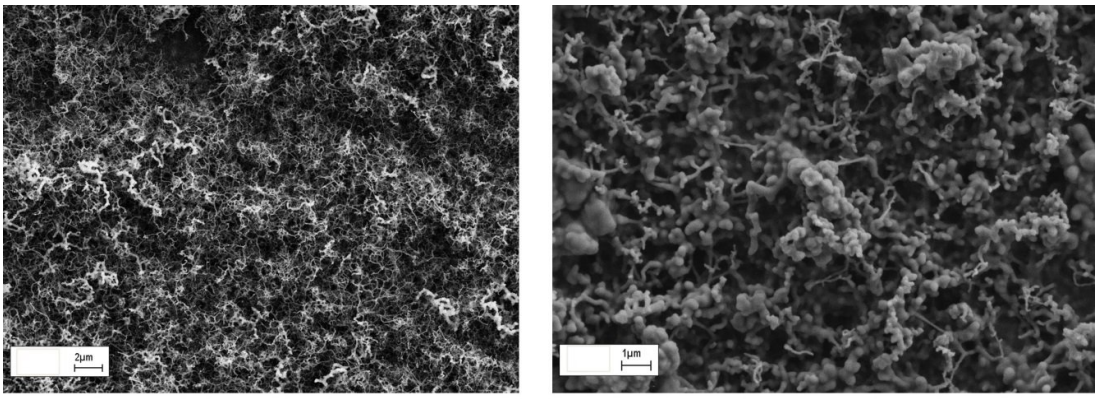

Figure 18 - Different Lumicyano 4% polymer formations under atmospheric conditions on different oily surfaces, showing (a) the densely-packed microfibrils (×10K) and (b) the spherical polymer condensing on the fibril formations (×20K).

There were two obvious exceptions to this predominantly noodle-like morphology in this Lumicyano (atmospheric) sample group. The first was observed on a sample extracted from a source that had previously exhibited fibrous formations (Figure 18a). Noodles in this latter fingermark were notably absent in comparison. Here, the polymer condensed into fused, flattened (two-dimensional) deposits, with an array of microfibrils appearing towards the edges in some areas of the sample (Figure 19a). The other exception was a print developed on a PE bag, which saw globules preferentially formed. The spherical structures varied in size, and generally comprised an underlying deposit of smaller ~0.3 µm spherical polymer and larger, less spherical grains of ~1-3 µm across (Figure 20b). A few scattered microfibrils and larger noodles were also present over this prevalently granular surface. This indicated that although Lumicyano 4% (atmospheric) preferentially deposited in fibrous formations, the result was not ubiquitous and may be possible due to the compression of the fibrils.

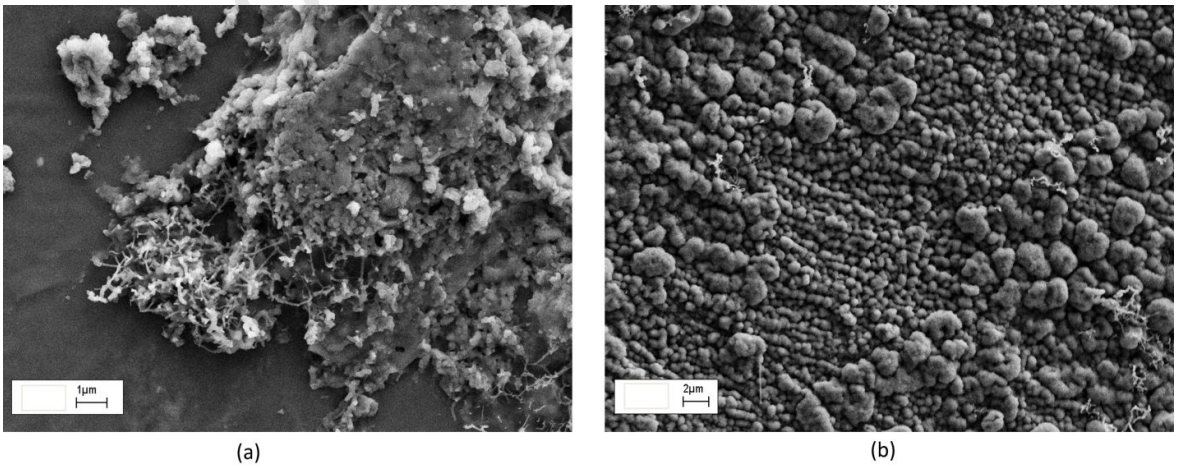

Figure 19 - The exceptions for Lumicyano 4% atmospheric: Non-noodle-like polymer structures on (a) an oily metallised plastic film (×20K) and (b) a clean PE bag (×8.4K).

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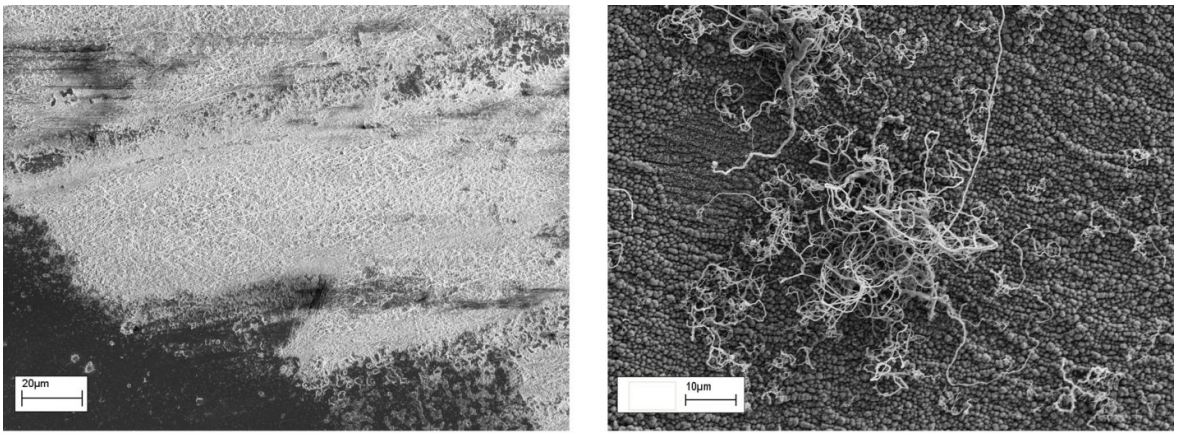

Figure 20 - Differential polymer formation on 'clean' surfaces, showing the predominant (a) noodles on a metallised plastic film (×2.5K) and (b) globular formations on a PE bin bag (×3.3K)

(3, 4) CA/BY40 and Lumicyano 4% vacuum (Trial 1 Process B and Trial 2 Process B 1st treatment)

SEM images demonstrated that both cyanoacrylate-BY40 and Lumicyano resulted in a similarly structured polymer deposit when exposed to vacuum conditions during development. The polymer consistently condensed into a granular film of fused and partially-fused polymer spheres, with no noodles or fibrils observed. The spherical globules varied in size and typically ranged between 0.2-0.4 µm in cyanoacrylate-BY40 fingermarks, and 2-4 µm in the Lumicyano-developed prints (Figure 21). Fusion, however, was mostly poorer in Lumicyano samples; therefore demonstrating that both polymer developers produced equally well-developed prints. Smaller grain-size and poorer fusion of the polymers both result in effective scattering of light. There was, furthermore, no noticeable difference between polymer formations on oily and clean prints.

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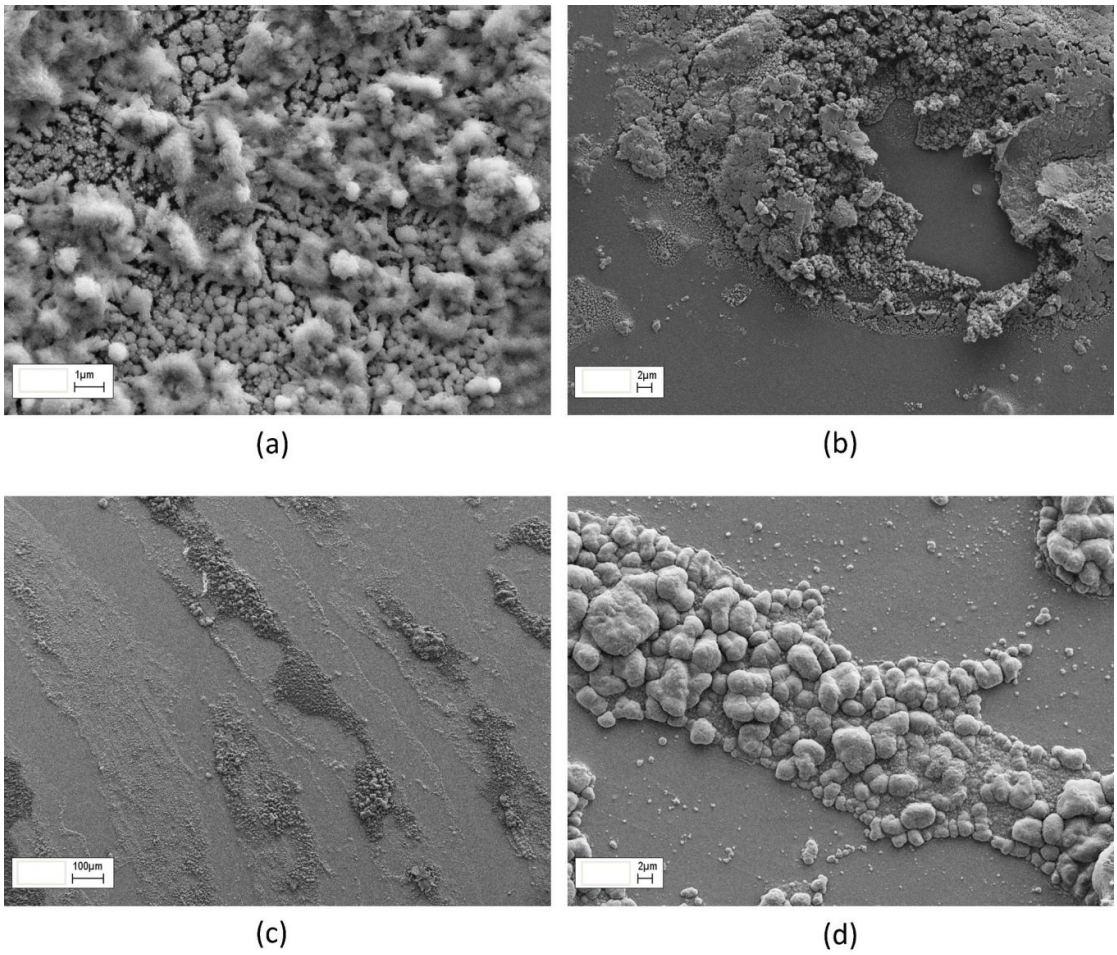

Figure 21 - Examples of CA/BY40 (a, b) and Lumicyano 4% (c, d) enhanced fingermarks under vacuum conditions, showing partial fusion of the spherical polymer.

(5, 6) Lumicyano 4% atmospheric – Lumicyano 4% atmospheric – BY40 (Trial 2 process A) & Lumicyano 4% vacuum – Lumicyano 4% atmospheric - BY40 (Trial 2 Process B)

Although only 3 samples of each double-fuming process with BY40 were examined, similar structures in the deposited polymer were observed which comprised a vast array of fibrous noodle formations (Figure 22). These, at times, appeared more dense and compacted, potentially indicating compression on handling/in transit post development. Other areas within the samples were seen to have deposited over a granular array; alternatively contained spherical particles forming on the tips of the noodles. These spherical polymers predominated in samples which had first been fumed under vacuum. The similarity between the two sets of results however was presumed to be a result of the final fuming process – Lumicyano 4% atmospheric – which denoted the preferential formation of noodles, described earlier. As SEM imaging in this analysis only looked at surface features, underlying structures could only be observed through voids in these surface features, where present, and did not address any stratigraphy.

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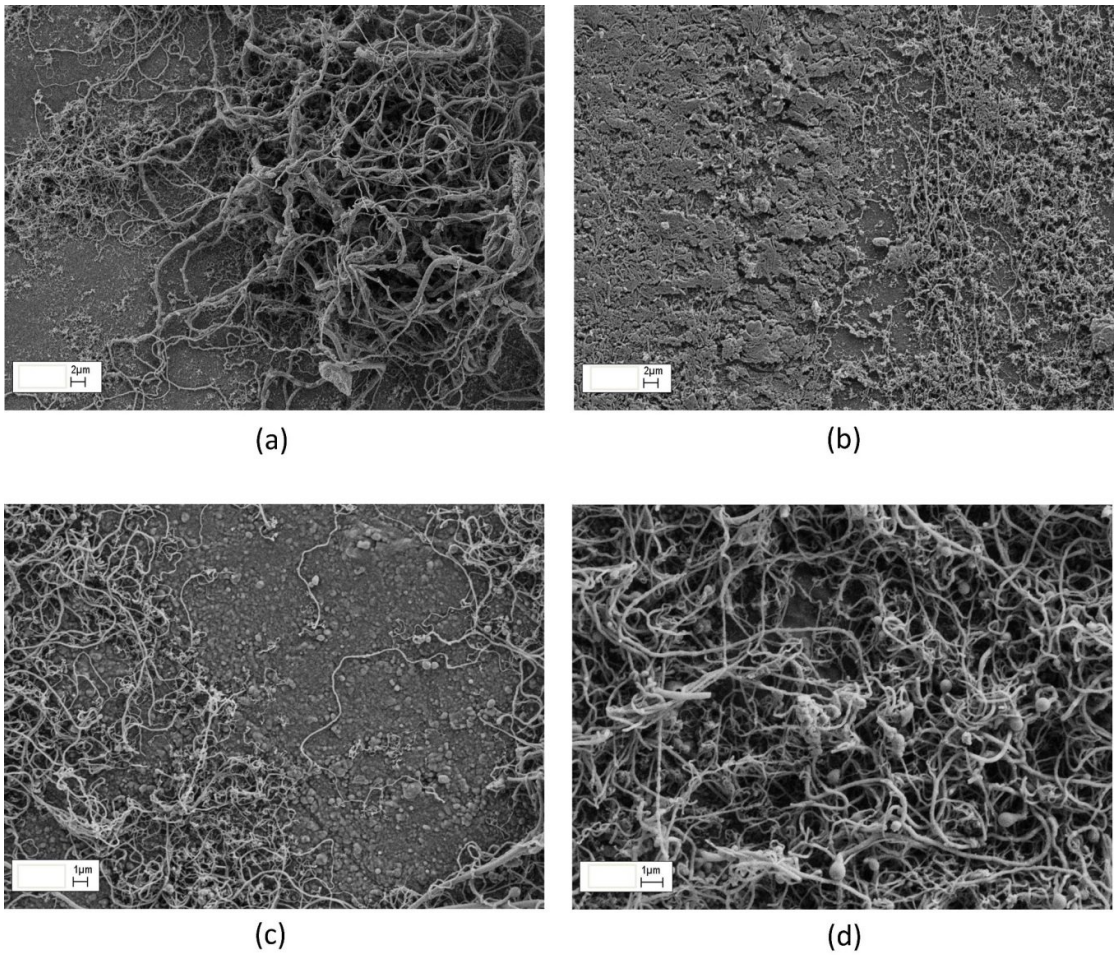

Figure 22 - The double-fuming process, showing: (a) the ranging noodle size and (b) the compressed polymer [left] in the Lumicyano 4% atmospheric-atmospheric sequence; (c) the accretions of spherical polymer formations underneath the fibrous polymer and (d) the capsules on the fibrous polymer also in the Lumicyano 4% vacuum-atmospheric sequence

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Conclusion

The results from this study demonstrate that the atmospheric/humidity process is superior to the vacuum process for both the two-step and one-step cyanoacrylate fuming. Although this correlates with previous research; it was found that staining with BY40 may still be possible after vacuum fuming (trial 1).

The sequences from trial 2 indicated that the first treatment with Lumicyano 4% at atmospheric conditions acted as an activation point for weak and undetected marks in the second treatment with Lumicyano 4% at atmospheric conditions. This unexpected result was found to be a result of the Lumicyano cyanoacrylate polymer growth in the z-direction and the targeting of previous cyanoacrylate deposits. According to the manufacturer guidelines, cabinets must be kept clean since "old cyanoacrylate residues will attract Lumicyano Powder fluorescence" which can diminish the operational effectiveness of the process and as a general rule, when using Lumicyano, the fuming cabinet should be cleaned in between each cycle. This helps to explain why the double process of Lumicyano under atmospheric/humidity conditions resulted in a significant number of new marks being detected after the second process.

Trial 3 demonstrates that the success of the double Lumicyano process is not due to the amount of cyanoacrylate or the fuming time but that the break in the two fuming cycles appears to be an important factor. Furthermore, trial 4 confirms that the double process is only successful for the Lumicyano polymer and not conventional cyanoacrylate. The increase in the number of marks from the sequence Lumicyano 4% vacuum - Lumicyano 4% atmospheric demonstrates that the use of vacuum cyanoacrylate fuming does not affect subsequent cyanoacrylate fuming with atmospheric/humidity conditions. SEM images have demonstrated that the polymer morphology in the sequence of vacuum-atmospheric changes from small granular structures to a fibrous/'noodle-like' structure which enables better light scattering and uptake of dye molecules.

Lumicyano fuming at atmospheric/humidity conditions provided a significantly higher detection rate when compared to vacuum fuming and both fuming conditions yielded marks with good ridge detail. Furthermore, the Lumicyano 4% fluorescence decays much faster under vacuum conditions (after 1 day) when compared to atmospheric/humidity fuming (up to 4 weeks), although both conditions provided minimal background fluorescence. The double Lumicyano process at atmospheric conditions appears to be the most effective process as it provides a significant number of new marks after the second treatment of Lumicyano. The sequence of

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Lumicyano under vacuum conditions followed by atmospheric/humidity fuming provided a high detection rate overall; however, the first process under vacuum conditions yielded a low number of marks which then increased significantly after the second process at atmospheric/humidity conditions. Nonetheless, vacuum fuming may have certain operational advantages as it was effective at developing latent marks on areas not directly exposed to the cyanoacrylate fumes. In general, this study has shown the superiority of the atmospheric/humidity method for cyanoacrylate fuming; however, further research into vacuum fuming is necessary to better understand the process and to contribute to the advancement of novel fuming methods. Future work will assess the use of other one-step processes such as Polycyano, PECA Multiband and Fuming Orange but assisted heating may be required due to the higher boiling point of these cyanoacrylate derivatives.

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Table 1 – SEM analysis: development conditions and number of samples.

Development conditions	Samples analysed
(1) Cyanoacrylate BY40 atmospheric (Trial 1 – Process A)	5
(2) Lumicyano 4% atmospheric (Trial 2 – Process A)	6
(3) Cyanoacrylate BY40 vacuum (Trial 1 – Process B)	6
(4) Lumicyano 4% vacuum (Trial 2 – Process B)	6
(5) Lumicyano 4% atmospheric – Lumicyano 4% atmospheric - BY40 (Trial 2 – Process A)	3
(6) Lumicyano 4% vacuum – Lumicyano 4% atmospheric - BY40 (Trial 2 – Process B)	3

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Table 2 – Cumulative number of latent marks detected by each process in trial 4.

Process A	No. of marks	Process B	No. of marks
CA-Atm Visible	60	CA-Atm Visible	67
BY40 Fluorescence	74	CA-Atm Visible	71
		BY40 Fluorescence	84

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